Deposition of durable wide-band silver mirror coatings using long-throw, low-pressure, DC-pulsed magnetron sputtering

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ABSTRACT

Sputter deposition at long-throw distances (15–30 in.) and low pressures (<1mTorr) were developed mainly for the semi-conductor industry to deposit metals and dielectrics into trenches or vias on silicon and gallium arsenide wafers. Scientists found that sputter depositions performed at pressures below 1mTorr (0.13 Pa) results in a virtually collision-free trajectory of the sputtered atoms from the target to the substrate. If the throw distance (source to substrate) is increased at these low pressures, the activated (ionized) gas and target atoms maintain their energy. We used this methodology along with dc-pulsed sputtering to deliver additional energy at the substrate. This allowed us to coat large optics (>21-in. diameter) in a standard box coater using smaller-diameter sputter cathodes. This paper will discuss the process used to successfully coat a 22-in.-diameter optic for the Keck Telescope in Hawaii with a new Wide-Band Durable Silver Mirror. The process uses smaller-diameter sputter cathodes in a 4-ft.-×-4-ft.-×-5-ft. box coater. We will also discuss how the process can be scaled to 36-in. or larger optics for use on terrestrial or space-based platforms.

Keywords: Long throw sputter deposition, dc pulsed sputtering, low pressure, durable silver mirrors

1. INTRODUCTION

Our goal was to develop a sputter process to deposit a durable, multilayer (17 layers) silver mirror on large optics (20–36-in. diameter) in a 4-ft.-×-4-ft.-×-5-ft. box coater. Commercial dc magnetron sputtering of metals, oxides, and nitrides in most systems takes place at a source-to-substrate distance somewhere between 3–6 in. and pressures in the 1–5-mTorr range.

The gas pressure in a sputtering system is relatively high compared with that in an evaporation system (10^{-3} mbar vs 10^{-6} mbar); therefore, the mean free path of a sputtered particle is three orders of magnitude less than a particle in an evaporation system. The sputtered target atoms and molecules are energetic as a result of the momentum transferred when struck by argon gas atoms and ions. For the target atoms to carry this energy to the substrate and not lose much of it through collisions, the distance between the target and the substrate must be as short as possible. This is normally why the substrate must be placed closer to the target in a sputter system than it is in an evaporation system.

Rossnagel[1] explains that in the semiconductor industry, pressures in the 0.1–0.5-mTorr range and source-to-substrate distances on the order of 12 in. are used to deposit metals and dielectrics into vias or channels in silicon and germanium substrates. Using this geometry simulates electron beam evaporation as far as coating distribution is concerned; the low gas pressure allows a long mean free path between collisions and allows the sputtered particles to maintain high energy.

We decided to take advantage of this methodology and design a new sputter deposition system that could sputter-deposit onto large optics (36-in. diameter) in a standard box coater. We used the following fundamentals to design our system:

1. Long throw (source to substrate); our 6-in.-diameter sputter gun looks like a point source for good uniformity over large areas.
2. Low pressure (0.1–0.5 mTorr); long mean free path and very few collisions of sputtered particles for 16–20 in.

3. New sputter cathodes, which utilize high-gauss magnets, help allow the cathode to run at low pressure (1.0×10^{-4}).
4. Gas inlet design distribution geometry.
5. New sputter deposition technology (dc-pulsed sputtering).

An in-house modeling program was used to calculate the placement of the sputter cathodes in the box coater to allow the best deposition coverage (uniformity) of our substrates (22–36-in. diameter). The system consisted of a 4-ft.-×-4-ft.-×-5-ft. box coater pumped by two 2000-L/s turbo-drag pumps, an 1100-L/s standard turbo pump, and a CTI-400 cryo pump. Five 6-in.-diameter sputter cathodes were used to deposit the mirror coating. The size (6 in.) of the circular cathodes was not picked for any particular reason other than it is what was available when the process was originally started, although real estate in the chamber for much larger cathodes is limited.

Based on the results of single-layer depositions, the geometry-modeling program predicted two sputter cathodes were to be set at a distance of 20 in. from the substrate and would deposit the NiCr and Ag layers. The other three cathodes were placed 15 in. from the substrate and were used to deposit the SiO_2, SiNx, and Ta_2O_5.

Fig. 1 is a photograph of the new sputter system with a freshly-coated mirror for the Keck telescope collimator.

Fig. 1: Sputter system with coated mirror for Keck telescope collimator.

2. THIN FILM COATING DESIGN

The basic thin film coating design used on the Keck substrate is shown below and explained in more detail in a paper by N. Thomas and J. Wolfe.[2]

Substrate M_2 M_1 M_2 M_3 $(LH)^6$ L AIR

M_1 = Silver M_2 = NiCrNx M_3 = SiNx L= SiO2 H= Ta_2O_5

The silver is deposited in 100% argon gas. The NiCrNx and SiNx are deposited in 100% N_2 gas and the oxides are deposited in an Ar/O_2 mixture.

3. DC-PULSED MAGNETRON REACTIVE SPUTTERING[3]

Asymmetric bipolar pulsed dc magnetron reactive sputtering is the latest technology for the deposition of dielectric coatings. This technique is based on the addition of a reverse-voltage bias pulse to the normal dc waveform. This bias pulse, when implemented at a frequency high enough to exploit the mobility differences between the ions and electrons in the plasma, accentuates the sputtering of dielectric films that accumulate on the target surface and effectively eliminates target poisoning and arcing. Pulse frequency and duty cycle can be varied to optimize the process for a specific target material. This technique is especially appealing because it can be implemented on a single cathode. Asymmetric bipolar pulsed dc technology has proven to be particularly beneficial for the enhancement of the deposited films' qualities, film uniformity, and film characteristics (n, k). The additional ionization of the pulsed plasma results in a hotter (greater electron temperature), more chemically active plasma, which tends improve the consistency of the film chemistry. This ionization enhancement (1.2–4 times) comes from the high frequency and pulsing components of the waveforms.

Fig. 2 shows the uniformity of Ag across 22 in. when deposited at different source-to-substrate heights (static deposition) and with the cathode located 16-1/5 in. from the center of the machine. The deposition of the other materials used in the design also followed this pattern.

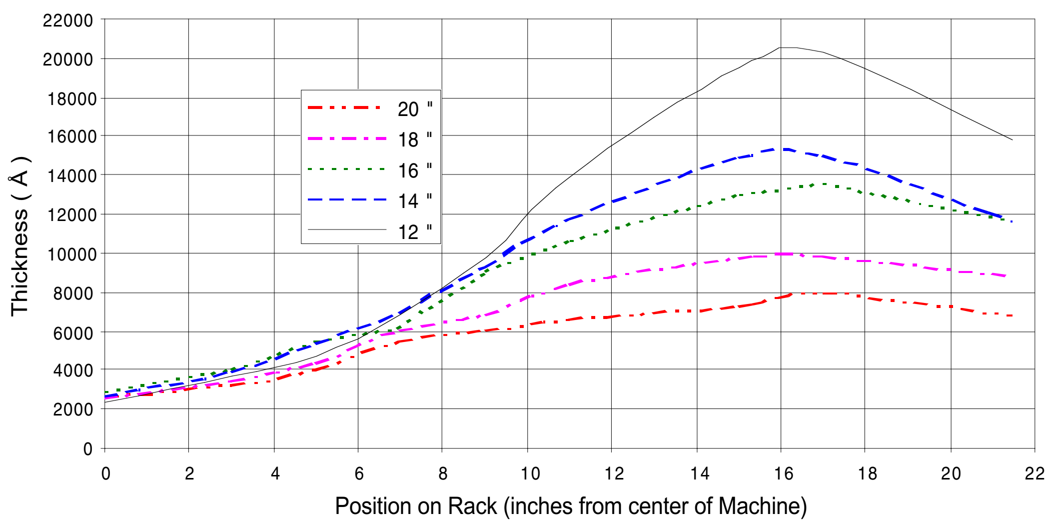

Fig. 2: 20, 18, 16, 12 inches, source to substrate. Target is silver (Ag).

4. EFFECT OF CHAMBER PRESSURE (MEAN FREE PATH) ON SiO_2 OPTICAL CONSTANTS (n, k)

It was shown that the chamber pressure used during the deposition of the SiO_2 layer had a large effect on the optical constants of the SiO_2 layers. This is caused by the decrease in ion bombardment at the substrate as the pressure is increased and the mean free path is decreased. The higher pressures cause a decrease in the packing density of the deposited film and implements voids throughout the film's microstructure, as described in the Introduction. We did not see this effect on the other oxides used in the designs.

Fig. 3 shows the relative mean free path as a function of pressure.

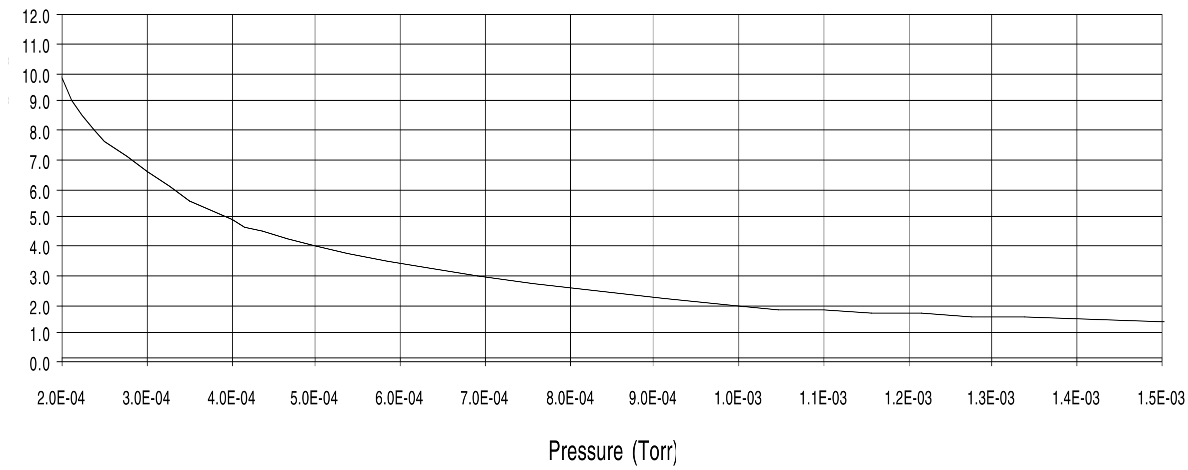

Fig. 3: Mean free path as function of pressure.

Fig. 4 shows the index of refraction vs wavelength of a SiO_2 film deposited with the same power and gas mix (Ar/O_2) while the pressure was changed by decreasing the pumping speed at the chamber, thus increasing the chamber pressure. The index of refraction of the film deposited at 1.5 mTorr is much lower than that deposited at 1 mTorr (the result of lower packing density and a voided film structure). The film deposited at 1.5 mTorr also had a higher extinction coefficient (absorption) than the film deposited at 1 mTorr.

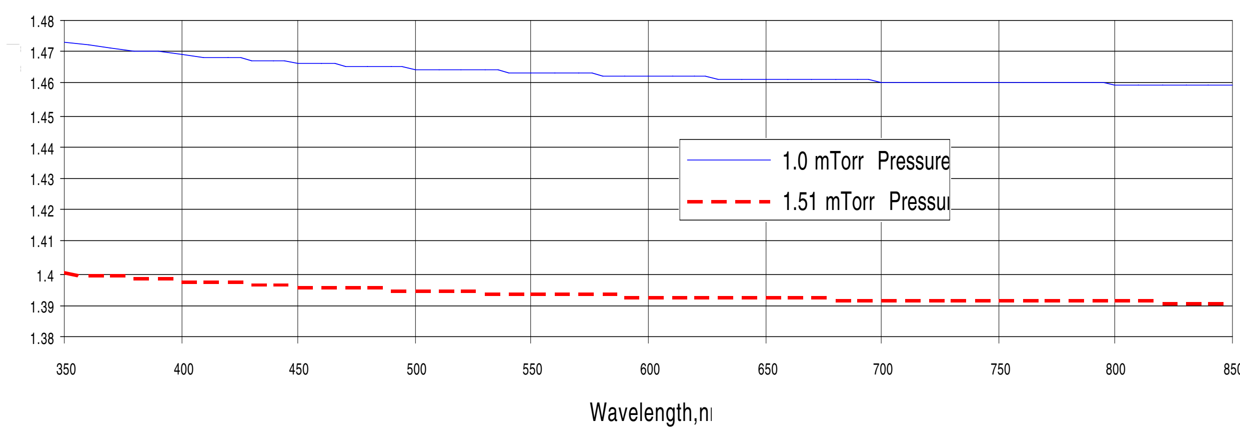

Fig. 4: SiO_2 at 1 mTorr vs 1.5 mTorr

Fig. 5 shows the extinction coefficient of the two films. The film deposited at 1 mTorr has 0% absorption while the film deposited at 1.5 mTorr has an extinction coefficient of $> 1.0 \times 10^{-3}$ at 350 nm.

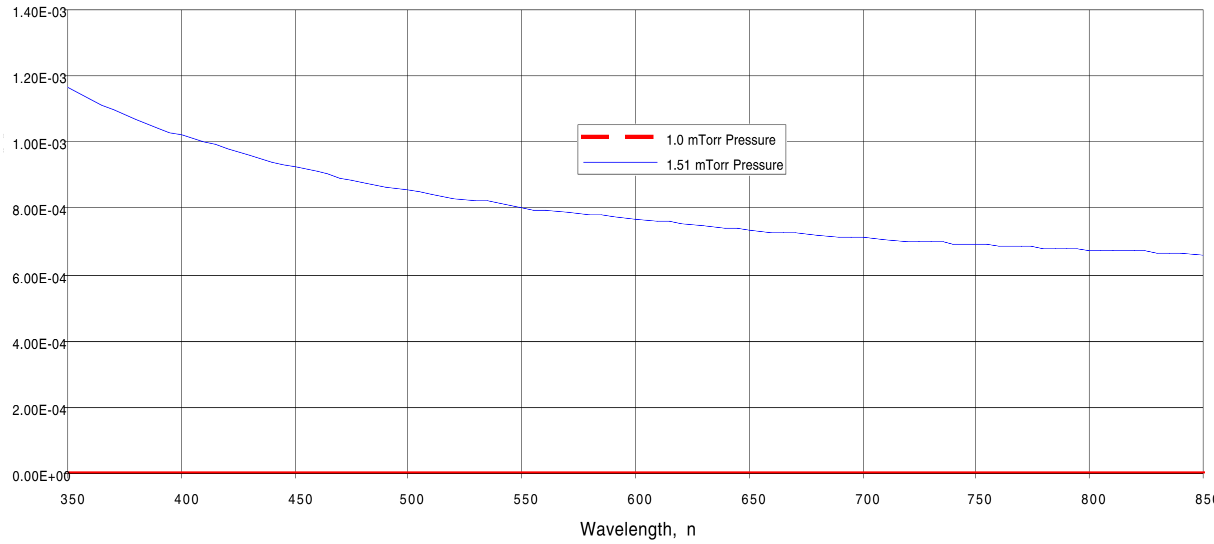

Fig. 5: SiO_2 1.0 mTorr vs 1.5 mTorr pressure.

Fig. 6 shows the n, k of a TiO_2 layer deposited at 0.5 mTorr.

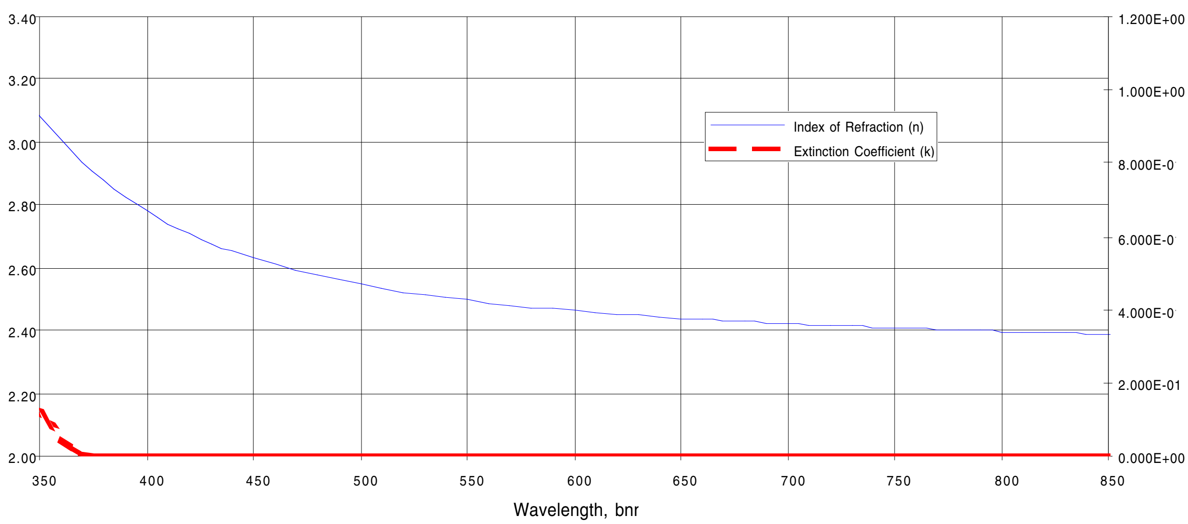

Fig. 6: Values for n and k of a TiO_2 layer deposited at 0.5 mTorr.

Fig. 7 shows the index value of a Ta_2O_5 layer deposited at 0.6 mTorr.

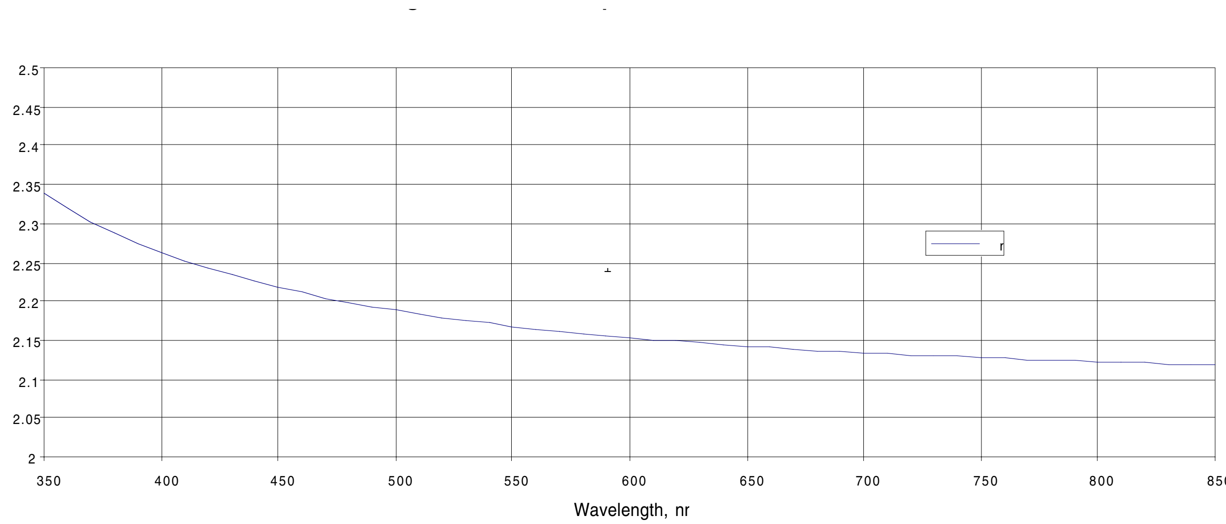

Fig. 7: Ta_2O_5 deposited at 0.6 mTorr.

5. FAMILY OF DURABLE SILVER MIRROR DESIGNS

We have designed and developed a family of durable silver mirror coatings. The designs vary only in percent reflection vs bandwidth and the quantity of layers.

Fig. 8 shows the family of durable silver coatings.

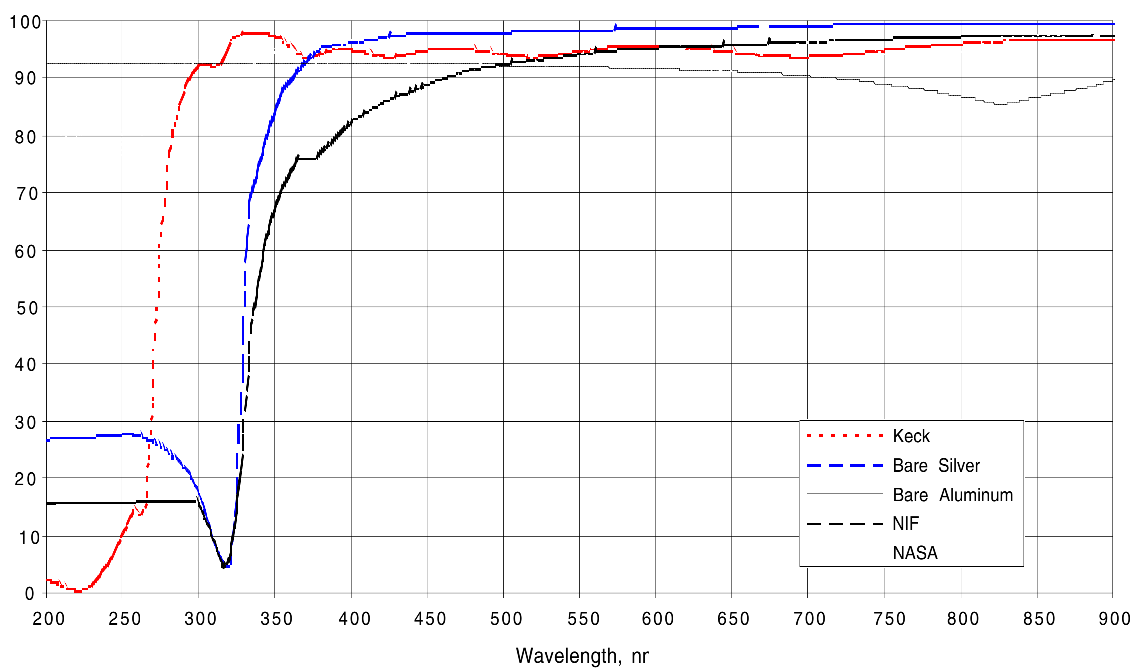

Fig. 8: Family of durable silver coatings.

Fig. 9 shows a durable silver design where the optical specifications were >97% reflectance in the range from 450–800 nm.

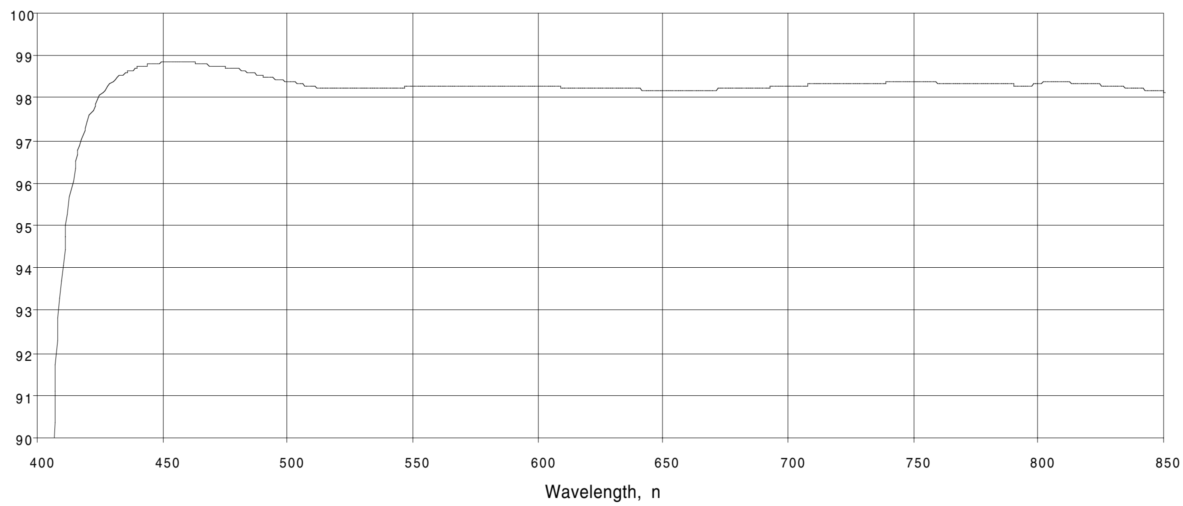

Fig. 9: Mirror with >97% reflectance.

Fig. 10 shows a theoretical design that was modeled for the Jet Propulsion Laboratory (JPL) and has a requirement of >98% reflectance from 400–1400 nm.

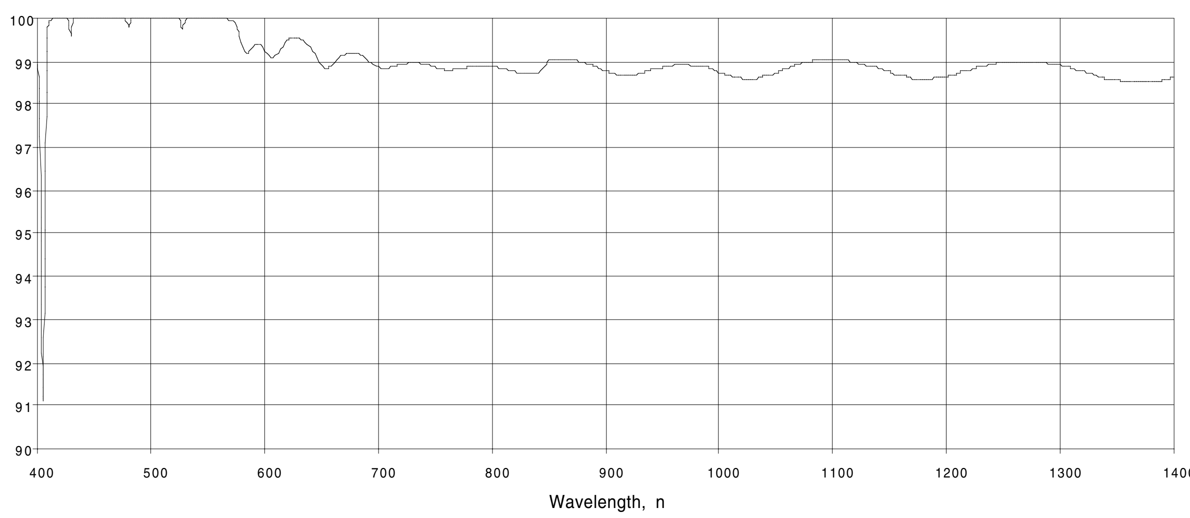

Fig. 10: JPL theoretical design.

6. ENVIRONMENTAL DURABILITY

Durable silver mirrors are capable of enduring the following environmental tests, which will degrade or destroy a standard enhanced silver or aluminum design.

6.1 Corrosion resistance

- Salt fog test

  72 hours in a 5% (NaCl) salt fog, at 95–98% relative humidity.

- Humidity test

  96 hours in a humidity cabinet at 60°C.

- Boiling salt water test

  1 hour submerged in boiling water with a 20% NaCl solution.

- Acid and base tests

  5 hours submerged in a 0.1n hydrochloric acid bath.
  5 hours submerged in a 0.1n sodium hydroxide bath.

- Hydrogen sulfide test

  The coating will pass 200 hours in a hydrogen sulfide atmosphere as per method 4.2 in ISO 9022-20.

6.2 Mechanical testing

- Coating adhesion test

  No evidence of coating removal when 3M-type tape is pressed firmly against the film and quickly removed (snapped) at a 90-deg angle to the coated surface.

- Crosshatch adhesion test (plastic substrates only)

  No evidence of coating removal when a crosshatch pattern is made on the coated surface using a razor blade. 3M-type tape is applied to the pattern and quickly removed at a 90-deg angle to the coated surface.

- Alcohol cheesecloth test

  Mil-Spec cheesecloth (#80) is saturated with ethyl alcohol and used with a crockmeter. Fifty passes (100 total) are made across the coated surface with a load of 200 g/cm^2.

- 20 rub eraser test

  The coating is tested by rubbing the coated surface with a standard eraser conforming to Mil-E-12397 mounted in the holding device. A force between 2 and 2-1/2 lb is applied. All strokes are made on one path for 20 strokes.

7. FUTURE DESIGNS

The coating designs discussed in this paper use SiO_2 as the low-index material. SiO_2 has an absorption band in the 8–10-µm region, and the film designs shown so far contain this absorption between 8–10 µm. The astronomical community would like to have a single coating that has high reflectivity across all wavelengths from 300 nm (UV) to 20 µm (IR). We plan to eliminate this absorption band by using another material such as MgF_2 for the low-index material. We may address this predicament using a plasma vapor deposition (PVD) process with ion-gun assist in future research and development of this project.

Fig. 11 shows a scan of the Keck design in the IR and the absorption in the 9–10 µm region.

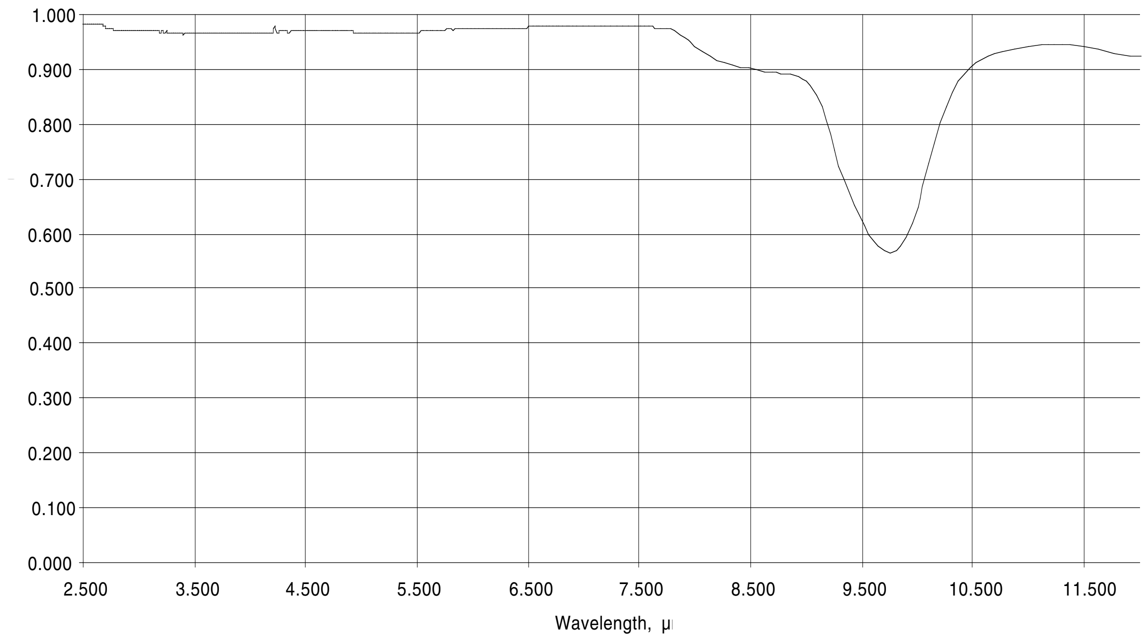

Fig. 11: LLNL-300 IR data measured by Dr. Michael Jacobson of Optical Data Associates.

8. CONCLUSIONS

By combining older with new sputter deposition technology, we have developed a novel process that allows sputter deposition on large optics in small chambers using smaller sputter guns. We have proven this technology by coating a 22-in. optic for the Keck Telescope in Hawaii with a new durable, high-reflecting Ag coating. The process can be utilized to deposit all metals, oxides, nitrides, etc. on large optics in small coating chambers using smaller sputter cathodes.

REFERENCES

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